(54) **Selective clogging of failed fibers**

Selektives Abdichten von defekten Fasern

Etanchement sélectif de fibres défectueuses

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Description

Field of The Invention

The present invention relates to the field of hollow fiber membranes. More particularly, the invention relates to a method of repairing hollow fiber membrane modules by the selective clogging of fibers which have failed during the manufacturing process.

BACKGROUND OF THE INVENTION

Membrane processes are material separation processes based upon the passage of material through the membranes wall to allow the selective separation of different materials.

A membrane is a solid configured as a thin layer of large surface area. Among other purposes, membranes serve as tools of separating two or more material components out of a mixture. The separative properties of membranes are based upon their different permeabilities to the various material components. Thus, in order to effect a separation, a driving force must exist across the membrane, which promotes the permeation of material through it, which may be a pressure or a concentration differential.

For the case of pressure differential driving force, the membrane must acquire the mechanical strength necessary to withstand the pressure differential. This contradicts the requirement that the membrane must be as thin as possible in order to allow large cross-flows. Therefore, the membrane is supported on a relatively porous support which sustains the mechanical load on the one hand, and allows free cross-flow of material through its large pore system on the other hand.

The necessity to have thin membrane layers, large surface areas in a given volume of a membrane separator, and a porous mechanical support has brought about the development of the hollow fiber membranes. These are thin tubes of circular cross-section. As such, they are self-supporting and occupy a small volume per unit surface area. A hollow fiber membrane module for separation is most frequently designed as a shell-and-tube assembly, as shown in Fig. 1. In such an arrangement, separate accesses to the interior (bore) and exterior (shell) sides of the HF membrane are possible, so as to enable the separation of the three major material flows: the feed, the reject which is collected at the opposite side of the module but at the same membrane face at which the feed was introduced, and the permeate which is the result of the flow through the membrane.

A bundle of hollow fiber (HF) membranes, when assembled in a shell-and-tube configuration for separation purposes, may contain a few broken or pinholed fibers. This is schematically illustrated by numeral 1 in Fig. 2, where a broken fiber is shown. The material transfer through these defects is non-selective and very high in comparison to the rest of the membrane wall, which was designed to acquire a desired selectivity. Therefore, such defects destroy the separative properties of the membrane bundle, and, if they are frequent, practically render it ineffective. It is therefore clear that, in order to be able to exploit fiber bundles containing failed fibers, it is necessary to find a method by means of which these defects can be repaired or avoided, or their undesirable result excluded.

The obvious method to avoid such defects is to improve the various steps of the process of membrane production. However, spinning hollow membrane fibers at an industrial scale always suffer from some extent of defects. This is especially so in the case of asymmetric membranes which have a very thin and thus vulnerable active skin, whereas the occurrence of pinholes of various diameters is very likely. In fact, the limit to thinning the skin in hollow fibers is set by the formation of defects.

Other occurrences of pinhole formation in membranes result not in the course of membrane production, but in the course of subsequent handling and treatments. This is particularly abundant in membranes of great brittleness and fragility, as is the case with ceramic and carbon membranes, and certain stiff, glassy polymers. Under such circumstances, the hollow fibers produced by the spinning system may be free of defects, but further handling which asserts mechanical stress, such as spool winding and unwinding, cutting the fibers to the length, inserting the hollow fiber bundle into the tube and end potting may cause damage.

The Prior Art

In order to overcome such problems, various methods of mending defects in hollow fiber membranes have been developed. In U.S. Patent No. 4,230,463 (Henis et al.), a method of coating a hollow fiber aimed at gas separation, with a silicone rubber layer which could clog the defects but, being highly permeable to gases, did not impede the total gas flow through the membrane to a significant extent. The coating polymer was applied as a solution in a volatile solvent such as pentane, and vacuum was applied at the bore side. By this procedure the polymer solution was forced into the defects and insured their clogging.

Alternatively, the polymer coating solution is introduced through the bore side of the hollow fiber while the active skin is at the shell side. Thus, the solution dries out upon reaching the outer side of the membrane, and a polymer clog forms selectively at the pinhole outlets to the shell side, where solvent evaporation takes place.

The abovementioned prior art processes are useful for treating defects not larger than 1 micron (and probably less), but are unsuitable for dealing with broken fibers.

An alternative method of avoiding defective membranes is individual detection and removal. If performed manually, this would be prohibitively time-consuming at

an industrial scale.

Other methods for the selective elimination of failed fibers are disclosed, for example in US-A-4,170,695; US-A-4,226,921; US-A-4,248,648 and WO-A-93,08987.

US-A-4,226,921, in particular, discloses drawing a sealant into the fibers by capillary attraction, but also to lower, if desired, the pressure exterior to the fibers, relative to the pressure on the sealant, to speed up the sealant's flow. However, while the lowered pressure will pass into the failed fibers through their failures, it will also pass to a considerable extent into the good fibers, as the gases present in them (nitrogen, oxygen), are significantly permeant to the molecular sieve membranes. Therefore the sealant rises up in the good fibers as well, and many of them become clogged, resulting in loss of module permeation capacity.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a method by means of which it is possible to carry out the selective elimination of failed fibers *after* potting in a bundle. The term "failed fibers" means hollow fibers that are broken, pinholed, injured, etc. Common to all these defects is the existence of a free passage between the feed and permeate sides, through which non-selective flow is enabled.

In particular, it is an object of the invention to solve the problem of selectively clogging the failed fibers while preventing the clogging of the non-failed (good) fibers.

In this way there are provided a simple and inexpensive method by means of which it is possible to use membrane modules which have lost their selectivity due to mechanical failure and through which it possible to manufacture hollow fiber bundles at an industrially acceptable production rate, avoiding the need for manual inspection and correction of mechanical failures.

The method of selectively clogging failed hollow fibers of a membrane module according to the invention, wherein the said fibers are packed together with a plurality of non-failed fibers in an arrangement that enables separate accesses to the shell and bore sides of the hollow fibers, comprises the steps of:

1. Clogging all fiber bores at one end of the pack or bundle of fibers;
2. Substantially evacuating the module both at the shell and at the bore side;
3. Filling the bore and the shell sides with a non-permeant gas
4. Holding the module vertically, disconnecting the bore side of the module from the non-permeant gas supply and dipping the module end opposite the clogged end into a reservoir containing a liquid sealant;
5. Applying a reduced pressure to the shell side for a period of time sufficient to permit suction of seal-

ant into the bores of failed fibers through the failure of which vacuum is applied to the bore side;
6. Allowing the sealant to solidify at least partially;
7. Cutting off a length of the fiber bundle such that non-failed fibers bores are exposed while failed fibers remain clogged;
8. If necessary, repeating the above steps at the other end of the module.

As used herein the term "vacuum" is meant to signify a reduced pressure, and includes a partial or a full vacuum, as the case may be.

It is worth noting that in stage 5 above, the sealant penetration into the failed fibers is much deeper than that into the perfect fibers which received no vacuum into their bores. Furthermore, stage 8 will normally be performed if the module is to have access to both hollow fiber ends of the bore sides.

According to another embodiment of the invention, the method comprises the steps of:

I. Clogging all fiber bores at one end of the pack or bundle of fibers;
II. Substantially evacuating the module both at the shell and at the bore side;
III. Filling the bore and the shell sides with a non-permeant gas
IV. Holding the module vertically, disconnecting the bore side of the module from the non-permeant gas supply and dipping the module end opposite the clogged end into a reservoir containing a liquid sealant;
V. Applying a reduced pressure to the shell side for a period of time sufficient to permit suction of sealant into the bores of failed fibers through the failure of which vacuum is applied to the bore side;
VI. Optionally, allowing the sealant to thicken, but not to solidify
VII. Filling the shell side with a permeant fluid, whereby to cause the permeant fluid permeating to the bore side to expel the sealant from non-failed fibers;
VIII. If necessary, repeating the above steps at the other end of the module.

The property used for the selective elimination which discriminates between failed fibers and good fibers is non-selective fluid transport between bore and shell sides of the failed fibers.

The selective elimination consists of introducing an adhesive (sealant) into the bores of the failed hollow fibers that hardens therein, thus clogging them and preventing the non-selective transport through them.

Brief Description of the Drawings

Figs. 1 through 5 illustrate various steps of the SCFF procedure;

Figs. 6 through 9 illustrate the auto-SCFF procedure;

Fig. 10 illustrates the pressure drops in the course of SCFF.

Detailed Description of Preferred Embodiments

The method according to one preferred embodiment of the invention is illustrated in Figs. 1 to 5.

Normal Potting Procedure.

In Figs. 1 and 2, two typical methods of the potting process are shown. In figure 1, potting is made by dipping the membrane module into the liquid sealant, thus clogging the bore ends. In figure 2, potting is made by applying the liquid, viscous sealant sideways at the module tube end while the HF bundle is extending out, thus leaving the HF ends open. For initiating the SCFF. the HF bundle must be brought to the position of figure 3A, namely, one end of the HF bundle should be open and the other end closed (Fig. 3B). In all Figures, only four hollow fibers are shown, for the sake of simplicity, which represent a bundle of a much greater number of fibers. In this figure and in some of the following figures as well, one hollow fiber is shown as broken, as indicated by numeral 1, and another is pinholed, as indicated by numeral 2, so that they generally represent failed fibers.

The module ends are potted by dipping the module in a liquid adhesive, generally indicated at 3 and 3'. The adhesive is cured by any known technique, such as by the lapse of time, or by other means such as contact catalysis or thermal setting. By this dipping method, the adhesive will most likely penetrate into all the hollow fiber (HF) bores to a certain depth (a few mm) and clog them. The fibers can then be opened by cutting the clogged portion, as shown at 4 in Fig. 3, which if it had no failed fibers, demonstrates a basic HF membrane module ready for assembly in a separation system.

In a slightly different method, potting is made by applying the adhesive to the side of the bundle near the tube end (Fig. 2), thus creating a thinner layer of adhesive 5 and 5', rather than by dipping the whole module end, as in Fig. 1 (rotation of the whole module about its axis is often applied, until adhesive is cured, in order to spread the adhesive across the tube). By the procedure illustrated in Fig. 2 the bore ends of the HF are left open.

Embodiment 1

Vacuum Only Treatment for Selective Clogging of Failed Fibers

In order to be able to operate according to the invention, a "tail" of certain length of the hollow fiber bundle must be provided at the extremities of the module, protruding out from the module tube, as is shown in Figs. 1 to 5.

It is essential that the HFs' bores at the other (non-treated) end are individually clogged. This implies that if the basic potting method was side adhesive application (Fig. 2), then the bore ends at the non-treated side should all be sealed off as is shown at 6 in Fig. 3B. Conversely, if both ends were sealed, it is possible to open-up one end by cutting off the sealed end, since, as stated, an extra length of the fiber is left at the extremities.

After the above-detailed situation has been obtained, the shell side 7 and the bore side 8 of the module are connected to a vacuum line to remove any existing fluid (Fig. 3A). A non-permeant is then introduced into the bore and shell sides, up to ambient pressure. By "non-permeant" is meant to indicate a gas that does not penetrate defect-free membrane walls.

The module is held vertically. The bore side is disconnected from the non-permeant gas supply, and immediately dipped into a liquid sealant 9, as shown in Fig. 4. The shell side 7 is then connected to a suction line which reduces the pressure at this side to a value Dp below that of the bore side (the bore side being at ambient pressure), for a preset time t, after which suction is released by introducing the non-permeant at the shell side. The preset time t is selected such that together with the bore diameter, the extent of vacuum (if partial), the sealant viscosity and its increase with time (the curing process), will lead to the desired penetration depth into the failed fibers. During the suction time, the sealant penetrates only into the bores of the failed fibers, since the pressure difference Dp is transmitted through the leak of the failed fiber from the shell to the bore side. This is illustrated by the raise of the sealant in the two failed fibers of Fig. 4, indicated by numerals 10 and 11.

Nominally, no sealant penetration is possible into the non-failed HFs, since the non-permeant filling them cannot be pumped out through the membrane walls. In practice, some penetration will take place into the non-failed fibers by capillary action due to mere contact with the sealant, but to a much lesser depth, as is shown by 12 in Fig. 4. The height of sealant penetration into the defective fiber bores is determined by controllable parameters, such as the preset suction (evacuation) time, the pressure differential Dp and the sealant viscosity.

After the seal is set within the HF bores, the bundle tail is cut at about point 13 (Fig. 4), to a length that removes the shallow seals formed at the ends of the non-failed fibers, but not the deeper seals that were formed along the defects of the fibers.

The resulting bundle shown in Fig. 5 has now selectively clogged failed fibers, as can be seen by comparison with the non-treated bundle of Fig. 3A.

If the finished module is of the type having a dead end at one of the bore sides, then the treatment terminating after the step shown in Fig. 5 is sufficient. How-

ever, if two bore side opening are desired, the tail-cutting step must be postponed. The module is turned upside down, the second module end is introduced into the sealant bath, suction is created to clog the other end of the failed fibers already clogged on the opposite end, and finally, the two tails are cut as described above.

Embodiment 2

Vacuum-Pressure Treatment for Selective Clogging of Failed Fibers

An important alternative to the suction release by the introduction of a non-penetrant, as described above, and the subsequent tail-cutting step, is the introduction of a penetrant at the shell side to release suction. Under such circumstances, the penetrant diffuses to the bore side, and increases the pressure therein above ambient pressure. In the non-failed fibers, this pressure increase pushes out the sealant from the fiber end, while in the case of failed fibers, the diffusing penetrant flows back to the shell side through the defect (pinhole or fracture), and thus it cannot apply a pressure within the fiber. By selecting the right timing of suction release by a pene-trant so that the sealant has thickened to the right extent, the process can be adjusted so as to open up the shallow seals from the non-failed fibers only but not from the failed fibers.

This embodiment of the invention is particularly important in the repairing of operative modules which have no "tail" ends, as specified in Embodiment 1, since the back diffusion of permeant does not call for tail cut-ting. This also greatly simplifies the membrane module design, since no provisions have to be made for the tail element and tail cutting. The situation obtained through this method is illustrated in Fig. 5, where the two non-failed ends 11 have been cleared of sealant by bore overpressure, formed by introducing a penetrant at the shell side.

Choice of Sealant

The sealant serving for the purpose of selective clogging of failed fibers (SCFF) should preferably be of the non-shrinking type. Therefore, sealants which solid-ify by solvent evaporation are less suitable. The pre-ferred materials are therefore those that set catalytically or thermally or in both ways, such as epoxy, acrylics, sil-icones, phenol-formaldehydes, furfuryl alcohol and the like. In terms of viscosity, which is a crucial parameter, determining the depth of sealant penetration into failed fibers, these materials may be divided into high and low viscosity groups.

To the first group belong partially polymerized adhesives having honey-like consistency, such as the epoxies and some silicones. The viscosities of sealants belonging to this group range between a few hundreds and a few ten-thousands of centipoise units. To the sec-ond group belong monomeric liquids having viscosities in the range of $3 \cdot 10^{-4} - 3 \cdot 10^{-3}$ Pa $\cdot$ s (0,3 - 3 cP) such as phenol-formaldehyde and furfuryl alcohol.

Recognizing that there are intermediate cases between these two groups, there is a span of 3 to 4 orders of magnitude in the viscosities of the two groups. Accordingly, two general methods of applying the seal-ant to the SCFF treatment process must be used, depending on whether the sealant has a high or a low viscosity.

It should further be noted that the invention is not limited to the use with any particular type of fiber, and can be employed, e.g., with carbon membranes just as with glass, ceramic or polymer membranes. The skilled person will be able to take into account construction material considerations, when choosing the most appropriate sealant for SCFF with a given membrane.

Process Parameter Analysis and Control

In this section a brief quantitative analysis of the SCFF parameters will be given. This analysis is pro-vided for a better understanding of the different varia-tions in procedures, which are claimed herein.

The most important parameter which has to be con-trolled is the depth of penetration of the sealant into the failed fibers. A shallow penetration will make it hard to define the crucial location of tail cutting (this has to be located beyond the depth of the sealant penetration into the perfect fibers, and below that of the failed fibers). On the other hand, too deep a penetration cannot be allowed:

1. If the defect in the membrane is close to the SCFF treated module end. In this case the pene-trating sealant may emerge from the defect to the shell side of the module, cement many fibers together and disturb the module functionality in var-ious ways.

2. If heat treatment is to be applied later to the mod-ule, since the sealant compound, which is typically an organic polymer, may be pyrolyzed, and the out-coming fumes may cause the clogged fiber to burst and severely to contaminate the entire membrane module. Such a sequence of events might be rele-vant with thermally stable membranes such as car-bon, ceramic and polyphosphazene-based membranes. Such membranes may have to be heat-treated to remove impurities which reduce permeation, or may be operated at high tempera-ture separation processes.

The factors which determine the sealant penetra-tion depth (PD) stem from laminar viscous flow consid-erations which govern the flow rate of the sealant into the fiber's bore. These factors are:

1. The pressure difference Dp between bore and

shell sides that is created during the suction of the sealant into the failed fibers' bores. This pressure difference provides the driving force for the sealant penetration into the failed fibers.

2. The suction duration.

3. The liquid sealant viscosity. Obviously, the lower the viscosity, the greater is the depth of penetration, everything else being equal.

4. The leak size at the defect points of the failed fibers, as shown in Fig. 10.

In the course of SCFF, the externally applied pressure difference Dp is divided into three components: $Dp1$, which falls on the leak at the defect point; the axial pressure drop Dpa which results from the non-penetrant viscous flow along the distance I between the leak and the surface of penetrating sealant, and Dps, which drives the sealant penetration. In Fig. 10, numeral 16 indicates the hollow fiber, numeral 17 the dead end of the fiber, numeral 18 is the shell compartment, 19 is the suction line and 20 is the sealant well.

We may therefore write:

$$Dp = Dp1 + Dpa + Dps \qquad (1)$$

5. The distance I which determines the magnitude of axial pressure drop Dpa, as described above. If the non-penetrant's viscosity is by far less than that of the sealant, this pressure drop may be neglected. This situation is possible when the non-penetrant is a gas which is at least 3 orders of magnitudes less viscous than the liquids (the sealers).

With regards to these arguments, we will assume in the following that factor no. 5, namely the axial pressure drop, can be neglected.

The basic equation of sealant flow into the hollow fiber (HF) obeys the Poisueille equation:

$$F = pDps \, r^4/(8 \times h) \qquad (2)$$

where F is the sealant flow rate in cm^3/sec into the HF, r is the inner HF radius in cm, assuming that its cross-section is circular; x is the depth of penetration of the sealant at any moment; and h is the viscosity in dyne-sec/cm^2 (poise).

The average axial liquid velocity dx/dt is obtained by dividing F by the tube cross-section πr^2 and rearranging. Thus:

$$dx/dt = r^2 Dp/(8h)x \qquad (3)$$

In the following, the description of the treatment will be split into 2 methods, for the high viscosity sealants.

(a) High viscosity, slow setting sealant

Based on eqns. (2)-(3), it can be readily shown that:

$$x = [2r^2 \, Dpt/(8h)]^{1/2} \qquad (4)$$

from which we find that, for instance, for a penetration depth (PD) of 5 cm, a high viscosity sealant of, say, 3 Pa·s (3000 cP) (group 1), an inner HF radius of 100 microns (.01 cm), and Dp 1 bar (10^6 dyne/cm^2 in cgs units), the penetration time will be, according to equation (4), 5^2X8X30/(2X10^{-4}X10^6) = 30 seconds. This penetration time falls within a range of the reasonably practical. Shorter times would be harder to control, while longer ones will cause catalytically set adhesives (epoxy) to become too viscous to advance to the desired depth into the fiber bores.

Accordingly, in one embodiment of the invention a high viscosity, slow setting sealant is applied to failed hollow fibers having an inner diameter of at least 10 μm, for a period of time of at least 0.1 minute, under a pressure difference Dp of at least 300 Pa (3 cm of water), until a penetration depth of at least 5 mm is achieved.

b. High viscosity, fast-setting sealant.

One may take advantage of using fast-setting adhesives to obtain the desired range of penetration depth independent of the application time of the driving force Dp. Explicitly, using the proper pressure drop and a given viscosity range of a freshly prepared, fast-setting adhesive, it is possible to apply the pressure drop for an excessively long time until the adhesive flow along the membrane bore ceases due to adhesive setting. This is a great simplification of the process.

Thus, in another preferred embodiment of the invention a high viscosity, fast setting sealant is applied to failed hollow fibers having an inner diameter of at least 10 μm, under a pressure difference Dp of at least 0.01 bar, for a period of time such that the desired penetration depth of at least 5 mm is achieved.

Embodiment 3

AUTO SCFF

Automatic SCFF is an SCFF operation effected integrally during the process of potting. The main difference between it and regular potting is by pre-exposing the module to a non-permeant for a sufficiently long period, so that the bore and feed sides are filled with it, as shown in Figs. 6 and 7. In Fig. 6 a carbon membrane is seen. As the non-penetrant SF_6 (sulfur hexafluoride) has been chosen, which flows from the tee connection downwards, so that the bottom side of the module is filled with it (SF_6 is much heavier than air so that it tends to settle down). In Fig. 7, the bottom connection of the sealant container 15, which was left open in the first stage (Fig. 6) is shut, and SF_6 flows upward the module to fill its upper parts.

In Fig. 8 the bung is removed from the container 15

and the sealant is poured into it. In Fig. 9, it is shown that the sealant container is positioned so that if the right amount of sealant is cast, it will fill the bottom shell side of the module up to the desired height due to the siphon effect and gravity. The filling level into the pores, proceeds up high into the failed fibers (higher than in the shell side, due to capillarity) since it can reject the non-permeant out to the shell side through the fracture/pin-hole opening. The penetration depth into the non-failed fibers proceeds only partially.

After curing, the module tail is cut at the right depth according to the regular procedure. As in the previously mentioned SCFF modes, the treatment is repeated at the other end of the module if two bore feed connections are desired.

As will be appreciated by the skilled person, the method of the invention can be used to correct a variety of defects, and is not limited to specific materials, sealants and bundle shapes. Many modifications can be effected in the methods employed, without exceeding the scope of the invention.

Claims

1. A method of selectively clogging failed hollow fibers (1, 2) of a membrane module packed together with a plurality of non-failed fibers in a shell-and-bore arrangement (7, 8), comprising the steps of:

   i. Clogging all fiber bores at one end of the pack or bundle of fibers;
   ii. Substantially evacuating the module both at the shell (7) and at the bore (8) side;
   iii. Filling the bore (8) and the shell (7) sides with a non-permeant gas
   iv. Holding the module vertically, disconnecting the bore side of the module from the non-permeant gas supply and dipping the module end opposite the clogged end into a reservoir containing a liquid sealant (9);
   v. Applying a reduced pressure to the shell side (7) for a period of time sufficient to permit suction of sealant (10, 11) into the bores of failed fibers through the failure of which vacuum is applied to the bore side (8);
   vi. Allowing the sealant to solidify at least partially;
   vii. Cutting off a length of the fiber bundle such that non-failed fibers bores are exposed while failed fibers remain clogged;
   viii. If necessary, repeating the above steps at the other end of the module.

2. A method of selectively clogging failed hollow fibers (1, 2) of a membrane module packed together with a plurality of non-failed fibers in a shell-and-bore arrangement (7, 8), comprising the steps of:

   i. Clogging all fiber bores at one end of the pack or bundle of fibers;
   ii. Substantially evacuating the module both at the shell (7) and at the bore (8) side;
   iii. Filling the bore (8) and the shell (7) sides with a non-permeant gas;
   iv. Holding the module vertically, disconnecting the bore side of the module from the non-permeant gas supply and dipping the module end opposite the clogged end into a reservoir containing a liquid sealant (9);
   v. Applying a reduced pressure to the shell side (7) for a period of time sufficient to permit suction of sealant (10, 11) into the bores of failed fibers through the failure of which vacuum is applied to the bore side;
   vi. Optionally, allowing the sealant to thicken, but not to solidify;
   vii. Filing the shell (7) side with a permeant fluid, whereby to cause the permeant fluid permeating to the bore (8) side to expel the sealant from non-failed fibers, thus opening them up;
   viii. If necessary, repeating the above steps at the other end of the module.

3. A method according to claim 1 or 2, wherein a slow setting sealant having honey-like consistency is applied to failed hollow fibers having an inner diameter of at least 10 μm, for a period of time of at least 0.1 minute, under a pressure difference D_p of at least 300 Pa (3 cm of water), until a penetration depth of at least 5 mm is achieved.

4. A method according to claim 1 or 2, wherein a fast setting sealant having honey-like consistency is applied to failed hollow fibers having an inner diameter of at least 10 μm, for a period of time of at least 0.1 minute, under a pressure difference D_p of at least 300 Pa (3 cm of water), until a penetration depth of at least 5 mm is achieved.

5. A method according to claim 1, wherein the non-permeant is SF_6.

6. A method according to any one of the preceding claims, wherein potting and selective clogging of failed fibers operations are carried out at the same time.

Patentansprüche

1. Verfahren zum selektiven Abdichten bzw. Verschließen von defekten Hohlfasern (1,2) eines Membranmoduls, die mit einer Vielzahl nicht defekter Fasern in einer Mantel-und-Bohrungsanordnung (7,8) zusammengepackt sind, mit den Schritten:

i. Abdichten bzw. Verschleißen aller Faserbohrungen an einem Ende des Packs oder Bündels von Fasern,

ii. im wesentlichen Evakuieren des Moduls sowohl an der Seite des Mantels (7) als auch an der Seite der Bohrung (8),

iii. Befüllen der Bohrungs- (8) und der Mantel-(7) Seiten mit einem nicht durchdringenden Gas,

iv. Senkrechtes Halten des Moduls, Trennen der Bohrungsseite des Moduls von der Zufuhr des nicht durchdringenden Gases und Eintauchen des Modulendes, das dem verschlossenen bzw. abgedichteten Ende gegenüberliegt, in ein ein flüssiges Dichtmittel (9) enthaltendes Reservoir,

v. Anwenden eines verringerten Drucks auf die Mantelseite (7) für eine ausreichende Zeit, um ein Einsaugen von Dichtmittel (10,11) in die Bohrungen von defekten Fasern zuzulassen, aufgrund deren Defekt sich Vakuum auf die Bohrungsseite (8) erstreckt,

vi. Zulassen, daß sich das Dichtmittel zumindest partiell verfestigt,

vii. Antrennen einer Länge des Faserbündels derart, daß nicht defekte Faserbohrungen frei liegen, während defekte Fasern verschlossen bleiben,

viii. falls erforderlich, Wiederholen der zuvor genannten Schritte am anderen Ende des Moduls.

2. Verfahren zum selektiven Andichten bzw. Verschließen defekter Hohlfasern (1,2) eines Membranmoduls, die mit einer Vielzahl nicht defekter Fasern in einer Mantel- und Bohrungsanordnung (7,8) zusammengepackt sind, mit den Schritten:

i. Abdichten bzw. Verschließen aller Faserbohrungen an einem Ende des Packs oder Bündels von Fasern,

ii. im wesentlichen Evakuieren des Moduls sowohl an der Seite des Mantels (7) als auch an der Seite der Bohrung (8),

iii. Befüllen der Bohrungs- (8) und der Mantel-(7) Seiten mit einem nicht durchdringenden Gas,

iv. Senkrechtes Halten des Moduls, Trennen der Bohrungsseite des Moduls von der Zufuhr des nicht durchdringenden Gases und Eintauchen des Modulendes, das dem verschlossenen bzw. abgedichteten Ende gegenüberliegt, in ein ein flüssiges Dichtmittel (9) enthaltendes Reservoir,

v. Anwenden eines verringerten Drucks auf die Mantelseite (7) für eine ausreichende Zeit, um ein Einsaugen von Dichtmittel (10,11) in die Bohrungen von defekten Fasern zuzulassen, aufgrund deren Defekt sich Vakuum auf die Bohrungsseite (8) erstreckt,

vi. optionales Zulassen, daß das Dichtmittel sich verdickt bzw. eindickt, aber nicht verfestigt,

vii. Befüllen der Mantelseite (7) mit einem durchdringenden Fluid, um das durchdringende Fluid zur Seite der Bohrung (8) hindurchtreten zu lassen, um das Dichtmittel aus den nicht defekten Fasern herauszudrücken und diese somit zu öffnen,

viii. falls erforderlich, Wiederholen der zuvor genannten Schritte am anderen Ende des Moduls.

3. Verfahren gemäß Anspruch 1 oder 2, wobei ein langsam abbindendes Dichtmittel (2) auf defekte Hohlfasern mit einem Innendurchmesser von mindestens 10 μm für eine Zeitspanne von mindestens 0,1 min und unter einem Druckunterschied Dp von mindestens 300 Pa (3 cm Wassersäule) angewendet wird, bis eine Eindringtiefe von mindestens 5 mm erreicht ist.

4. Verfahren gemäß Anspruch 1 oder 2, wobei ein schnell abbindendes Dichtmittel (2) mit einer honigartigen Konsistenz auf defekte Hohlfasern mit einem Innendurchmesser von mindestens 10 μm für eine Zeitspanne von mindestens 0,1 min und unter einem Druckunterschied Dp von mindestens 300 Pa (3 cm Wassersäule) angewendet wird, bis eine Eindringtiefe von mindestens 5 mm erreicht ist.

5. Verfahren gemäß Anspruch 1, wobei das nicht durchdringende (Gas) SF_6 ist.

6. Verfahren gemäß einem der vorstehenden Ansprüche, wobei die Arbeitsschritte beim Einbetten (potting) und selektiven Verschließen bzw. Abdichten defekter Fasern gleichzeitig ausgeführt werden.

Revendications

1. Procédé d'obturation sélective de fibres creuses

défectueuses (1, 2) d'un module à membrane, groupées ensemble avec plusieurs libres non défectueuses dans un agencement à enveloppe-et-lumières (7, 8), comprenant les étapes dans lesquelles :

i. on obture les lumières de toutes les fibres à une extrémité du groupement ou faisceau de fibres ;

ii. on évacue sensiblement le module à la fois du côté enveloppe (7) et du côté lumières (8) ;

iii. on remplit les côtés lumières (8) et enveloppe (7) d'un gaz ne passant pas par perméation ;

iv. on maintient le module verticalement, on isole le côté lumières du module de l'alimentation en gaz ne passant pas par perméation et on plonge l'extrémité du module opposée à l'extrémité obturée dans un réservoir contenant un agent liquide d'obturation étanche (9) ;

v. on applique une pression réduite au côté enveloppe (7) pendant une période de temps suffisante pour permettre l'introduction par aspiration de l'agent d'obturation étanche (10, 11) dans les lumières des fibres défectueuses à travers le défaut desquelles une dépression est appliquée au côté lumières (8) ;

vi. on permet une solidification au moins partielle de l'agent d'obturation étanche ;

vii. on sectionne une longueur du faisceau de libres de manière que des lumières de fibres non défectueuses soient mises à découvert, tandis que les libres défectueuses restent obturées ;

viii. si cela est nécessaire, on répète les étapes ci-dessus à l'autre extrémité du module.

2. Procédé d'obturation sélective de fibres creuses défectueuses (1, 2) d'un module à membrane, groupées ensemble avec plusieurs fibres non défectueuses dans un agencement à enveloppe-et-lumières (7, 8), comprenant les étapes dans lesquelles :

i. on obture les lumières de toutes les fibres à une extrémité du groupement ou faisceau de fibres ;

ii. on évacue sensiblement le module à la fois du côté enveloppe (7) et du côté lumières (8) ;

iii. on remplit les côtés lumières (8) et enveloppe (7) d'un gaz ne passant pas par perméation ;

iv. on maintient le module verticalement, on isole le côté lumières du module de l'alimentation en gaz ne passant pas par perméation et on plonge l'extrémité du module opposée à l'extrémité obturée dans un réservoir contenant un agent liquide d'obturation étanche (9) ;

v. on applique une pression réduite au côté enveloppe (7) pendant une période de temps suffisante pour permettre l'introduction par aspiration de l'agent d'obturation étanche (10, 11) dans les lumières des fibres défectueuses à travers le défaut desquelles une dépression est appliquée au côté lumières ;

vi. on permet optionnellement à l'agent d'obturation étanche d'épaissir, mais pas de se solidifier ;

vii. on remplit le côté enveloppe (7) d'un fluide passant par perméation, afin d'amener le fluide passant par perméation, qui passe par perméation du côté lumières (8), à expulser l'agent d'obturation étanche des fibres non défectueuses, les ouvrant ainsi ;

viii. si cela est nécessaire, on répète les étapes ci-dessus à l'autre extrémité du module.

3. Procédé selon la revendication 1 ou 2, dans lequel un agent d'obturation étanche prenant lentement, ayant une consistance analogue à du miel, est appliqué aux fibres creuses défectueuses ayant un diamètre intérieur d'au moins 10 µm, pendant une période de temps d'au moins 0,1 minute, sous une différence de pression D_p d'au moins 300 Pa (3 cm d'eau), jusqu'à ce qu'une profondeur de pénétration d'au moins 5 mm soit atteinte.

4. Procédé selon la revendication 1 ou 2, dans lequel un agent d'obturation étanche à prise rapide, ayant une consistance analogue à celle du miel, est appliqué à des fibres creuses défectueuses ayant un diamètre intérieur d'au moins 10 µm, pendant une période de temps d'au moins 0,1 minute, sous une différence de pression D_p d'au moins 300 Pa (3 cm d'eau), jusqu'à ce qu'une profondeur de pénétration d'au moins 5 mm soit atteinte.

5. Procédé selon la revendication 1, dans lequel la matière ne passant pas par perméation est du SF_6.

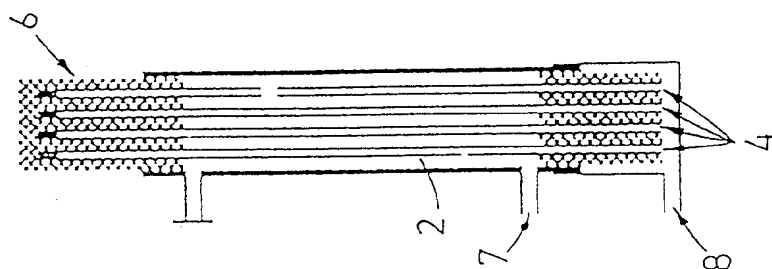6. Procédé selon l'une quelconque des revendications précédentes, dans lequel des opérations d'enrobage et d'obturation sélective des fibres défectueuses sont exécutées en même temps .
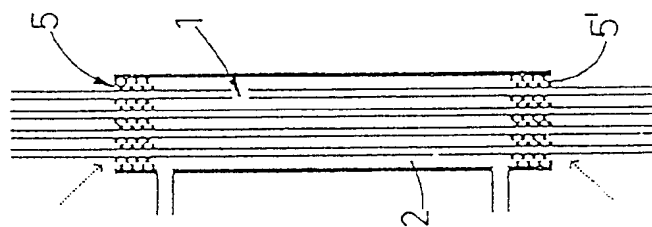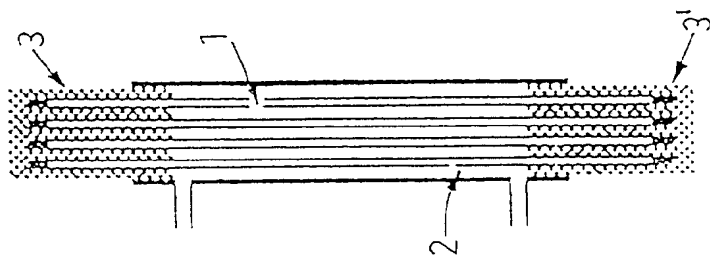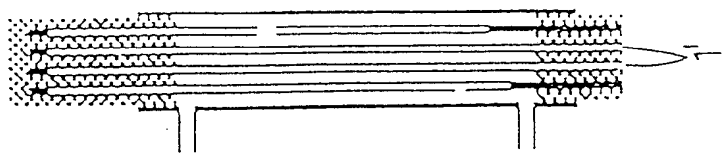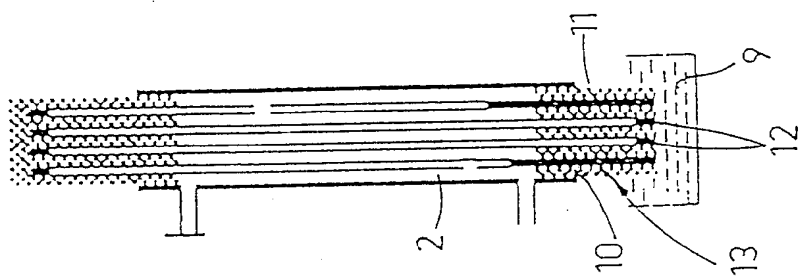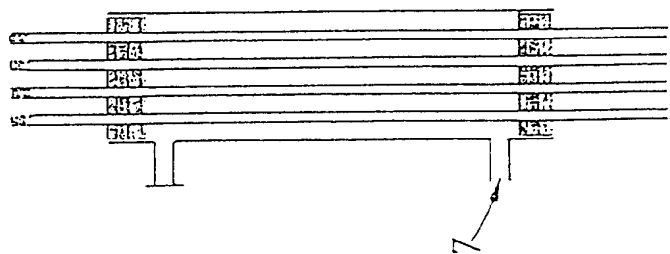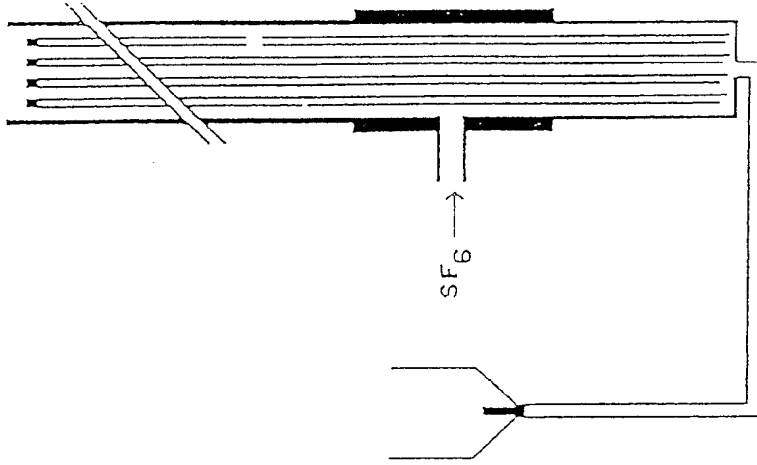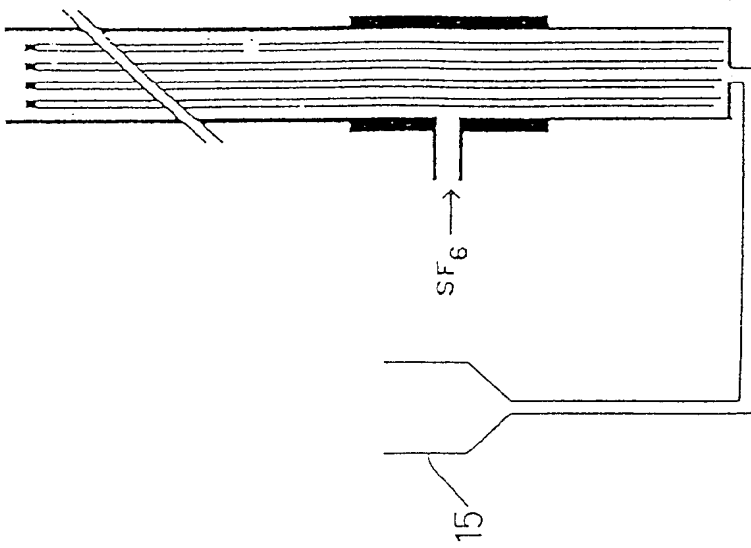
Fig. 3A

Fig. 2

Fig. 1

Fig. 5

Fig. 4

Fig.3B

Fig. 7

Fig. 6

Fig. 9

Fig. 8

Fig. 10